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*Short Communication*

# Electrochemical Corrosion Resistance of Carbon Steel Rebar in Concrete Structures Exposed to 3.5wt% NaCl Solution: Effect of Green Inhibitors and Micro-Silica as Partial Replacement

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In this research, the simultaneous effect of mineral admixtures and a green inhibitor on corrosion resistance of carbon steel rebar in concrete exposed to 3.5wt% NaCl solution were studied. Portland cement (PC) was replaced by a mixture of metakaolin and micro silica (MS) admixtures. Electrochemical impedance spectroscopy technique and polarization measurement, water absorption test and compressive strength were used to assess the corrosion resistance of carbon steel in concrete. The electrochemical results revealed that the MS15M15 concrete containing both MS and metakaolin had higher corrosion potential and resistance than the other samples. The water absorption results exhibited that the MS15M15 specimen significantly influence on mobility and penetration of water and chloride ions. The morphology characterization of the surface of carbon steel rebar showed that slight pits and low corrosion products appeared on the surface of MS15M15 sample which was according to the results achieved from electrochemical tests. The polarization results revealed that the disporopsispernyi leaves extract (DPLE) was a mixed-type inhibitor that improved the corrosion resistant performance in carbon steel rebar considerably with formation of an organic layer to prevent both cathodic and anodic corrosion reactions. These findings exhibited that mineral admixtures and DPLE inhibitors led to a decrease of hydroxide ion contents which caused an enhancement in passivation of carbon steel rebar and an enhanced corrosion resistance.

**Keywords:** Partial replacement; Carbon steel rebar; Mineral admixtures; Electrochemical corrosion behavior; Green inhibitor

## 1. INTRODUCTION

Corrosion behavior of carbon steel in concrete decreases the durability of concrete structure [1, 2]. Corrosion occurs regardless of the inherent ability of concrete to protect steel rebars. The corrosion is created by the penetration of corrosive ions or the loss of alkalinity of concrete [3]. The corrosion control techniques include the use of admixtures in the concrete cement, cathodic protection and surface treatments for the steel rebar [4, 5]. The use of additives alone is attractive because they are relatively inexpensive. Mineral additives are commonly used in numerous applications such as bridges, high performance concrete buildings and on-shore and off-shore structures [6, 7]. Well-known mineral additives contain fly ash, silica fume, and rice husk ash [8]. The use of these materials in production of concrete has positive environmental effects and minimizes problems regarding its disposal.

Khatib et al. [9, 10] indicated that the 20% of cement replacement by metakaolin has significantly increased the compressive strength of the mortar by 50%. Justice et al. [11] compared the effect of using two different types of metakaolin on the concrete workability and setting time. It was found that the addition of metakaolin causes a significant reduction in concrete workability and setting time.

Moreover, the use of micro-silica (MS) in concrete can be helpful in reducing corrosion rate [12]. Recently, many studies exhibited that silicate compositions can considerably decrease the mobility and penetration of chloride ions into concrete structures. Kulakowski et al. showed the effect of MS additions on the corrosion resistance of carbon steel in concrete containing varying MS content [13]. When MS concentration was more than 10%, the carbonation induced corrosion of steel rebar reduced. The use of micro- and nano-technology in concrete has improved the metal's corrosion behavior and optimized the chemical, physical and electrochemical properties of the structures. Feky et al. exhibited the corrosion resistance of reinforcement steel was enhanced with increasing nano-silica/potassium silicate-ratio which can be associated to the size of silica nanoparticles and the capability to fill up the pores in potassium-silicate coating [14].

Corrosion inhibitors are usually used in industries to decrease the corrosion rate of carbon steels [5]. However, most compounds are artificial chemicals that can be very expensive and dangerous for living organisms and the environment [6]. Thus, to develop environmentally friendly and most effective corrosion inhibitors, the synthesis of green corrosion inhibitors is very attractive among researchers [7]. Green inhibitors are available as essential oils or extracts. Different parts of plants have several compounds that meet these criteria, so plants are a potential source of novel corrosion inhibitors [8]. Numerous scientific studies were recently attained to consider the effect of some natural substances as green inhibitors on the corrosion of various metals, such as steel, in different corrosive media [9].

However, metakaolin and MS have been proven to improve compressive strength of concrete and decrease the volume-specific surface area and permeability, the simultaneous effect of metakaolin and MS on corrosion resistance of carbon steel rebar has not been reported before. Also, many studies have been done to evaluate the green corrosion inhibitors for carbon steels [15, 16], the disporopsispernyi leaves extract (DPLE) has never been investigated as an inhibitor of carbon steel corrosion in saline solution. Thus, this work focuses on the mechanical property and electrochemical corrosion behavior of carbon steel in concrete due to simultaneous effect of both additives and efficiency of a natural DPLE as corrosion inhibitor for carbon steel in 3.5 wt% NaCl solutions.

## 2. MATERIALS AND METHOD

In this work, concrete was produced under controlled conditions at room temperature using Portland cement (PC), coarse aggregates, fine and water. The cement replacement was done with metakaolin and micro silica (MS). Table 1 is shown the chemical composition of PC, MS and metakaolin. The water-cement ratio of the mixes was 0.5.

**Table 1.** Chemical composition of PC, metakaolin and MS

| | PC (wt%) | Metakaolin (wt%) | MS (wt%) |
|---|---|---|---|
| $SiO_2$ | 20.50 | 51.71 | 86.73 |
| $Al_2O_3$ | 4.89 | 42.98 | 0.00 |
| $Fe_2O_3$ | 3.12 | 0.96 | 0.99 |
| CaO | 64.15 | 0.24 | 2.54 |
| MgO | 2.02 | 0.17 | 7.53 |
| $K_2O$ | 0.67 | 0.15 | 3.13 |
| $Na_2O$ | 0.25 | 0.01 | 1.38 |
| $SO_3$ | 2.97 | 0.00 | 2.58 |
| LOI | 0.89 | 0.56 | 6.48 |

Table 2 reveals the composition of the various mixtures. The prepared mixtures were transferred into the cylinder mold with a diameter of 10 cm and height of 30 cm with 90% relative humidity at room temperature for 24 hours. In order to study the effect of combinations of MS and Metakaolin on corrosion resistance of the carbon steel rebars, electrochemical experiments were performed on carbon steel rebar into 3.5wt% NaCl solution as an corrosive environment in 8 weeks exposure time. Carbon steel rebar with a diameter of 1.2 cm and long of 10 cm was embedded in the cylinder. The chemical composition of used steel is exhibited in Table 3.

**Table 2.** Proportions of mixtures for various concrete structures

| Sample | PC (wt%) | Metakaolin (wt%) | MS (wt%) |
|---|---|---|---|
| PC | 100 | 0 | 0 |
| M30 | 70 | 30 | 0 |
| MS30 | 70 | 0 | 30 |
| MS15M15 | 70 | 15 | 15 |

**Table 3.** Composition of used carbon steel rebar (wt%)

| Carbon | Si | S | Mn | Cr | P | Ni | Fe |
|---|---|---|---|---|---|---|---|
| **0.17** | 0.25 | 0.018 | 0.46 | 0.15 | 0.0045 | 0.11 | Residual |

In order to make working electrodes, the concrete mixture was transferred into the cylindrical molds, while carbon steel rebars were positioned vertically in the center of cylinder mold. Open circuit potential was performed by a high-impedance voltmeter via an input resistance. The three-electrode electrochemical system was applied to study the corrosion resistance of carbon steel reinforcement in concretes. The carbon steel rebar in concrete, graphite and saturated calomel electrodes were used as a working, counter and reference electrodes, respectively. The analysis was recorded after immersion into 3.5wt% NaCl as a destructive environment. The EIS was done in a frequency range of 0.01Hz to 0.1MHz. The polarization test was done at 1mV/s scanning rate.

In order to study the effect of green inhibitors on corrosion behavior of carbon steel embedded in concrete, the disporopsispernyi leaves extract (DPL) was washed by DI water and dried at room temperature. Then it was successfully powdered and 8 g of powdered DPL were mixed by 0.5 L DI water in a magnetic induction stirrer at 70 °C for one hour. Then, the obtained solution was filtered by a filter paper. After that the solution was stirred once more at 70 °C for one day. The gel was then obtained with high viscosity and powdered. Finally, the obtained powder was applied as a corrosion inhibitor in all experiments. The tests were performed in 3.5 wt% NaCl solution with 0, 10, 50 and 100 mg/L of DPLE at room temperature.

For considering water absorption of mixes, cylinder-shaped samples with diameter of 10 cm and height of 20 cm were used according to ASTM C642. In order to determine the water absorption, the specimens were exposed to 3.5wt% NaCl solution. The water absorption of specimens was recorded after exposure time of one, two and four weeks. Compressive strength value was achieved according to the BS-1881 technique. Scanning electron microscope (SEM) was used to consider the surface morphology of the samples.

## 3. RESULTS AND DISCUSSION

Fig. 1 shows polarization plots of carbon steel rebar in different mixtures exposed to 3.5wt% NaCl solution after 2 months. As revealed in Figure 1, the anodic polarization plots are considered by passive regions in all carbon steel embedded in concrete, showing the passive layers have clearly formed on the surface of carbon steel when the steel rebar was exposed to 3.5wt% NaCl [17]. Furthermore, a significant shift was found in corrosion potential to a positive direction which reveals that the dissolution of anodic metal was capably retarded by changing the admixture content in concrete.

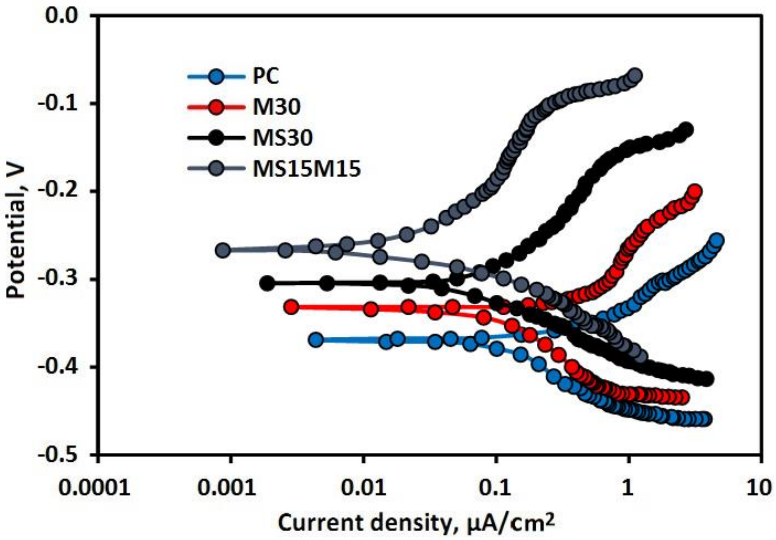


**Figure 1.** The polarization curves of carbon steel rebar in concretes with various mixtures exposed to 3.5wt% NaCl solution after 2 months at 1mV/s scanning rate at room temperature

**Table 4.** Corrosion factors attained from polarization plots in figure 1.

| Mixtures | Corrosion current density(μA/cm$^2$) | Corrosion potential (V) | $\beta_c$(mVdec$^{-1}$) | -$\beta_a$(mVdec$^{-1}$) |
|---|---|---|---|---|
| PC | 0.821 | -0.371 | 44 | 24 |
| M30 | 0.642 | -0.331 | 47 | 28 |
| MS30 | 0.086 | -0.304 | 49 | 31 |
| MS15M15 | 0.048 | -0.273 | 43 | 38 |

The passive zone for MS15M15 sample was much wider than the other specimens. Additionally, the passive current density for MS15M15 sample was lower than the other specimens, representing the enhanced corrosion behavior of carbon steel rebar in concrete containing both MS and metakaolin admixtures. It may be related to the metakaolin that reacted with free $Ca(OH)_2$ during the hydration process of PC and formed additional calcium silicate hydrate, which improved the mechanical property and durability in concrete [23]. Corrosion factors obtained from polarization plots in Fig. 1 are indicated in table 4.

The corrosion levels can be separated in four-levels presented by the Durar-Network Specification. However, the corrosion current density ($i_{corr}$) of the MS15M15 sample in 3.5wt% NaCl solution was lower than other samples. Therefore, except the PC and M30 specimens, the steel rebars in MS30 and MS15M15 admixtures remained in the passive state in the electrochemical corrosion process which exhibited high resistance to corrosion of carbon steel embedded in concrete in the marine environment.

The compressive strength of samples are shown in figure 2. By comparing the various replacement admixtures, it can be determined that at any exposure time the MS15M15 mixture has a greater strength. Increase in strength may be because of the high content of $Al_2O_3$ in metakaolin. It reacts both in pozzolanic and hydraulic methods which results in more pore and denser structure. It was also determined that MS15M15 mixture indicates higher strength compared to other specimens. Moreover, concrete with the MS showed a higher primary strength at each percentage MS30 replacement. The final strength for all admixtures was greater than the PC sample.

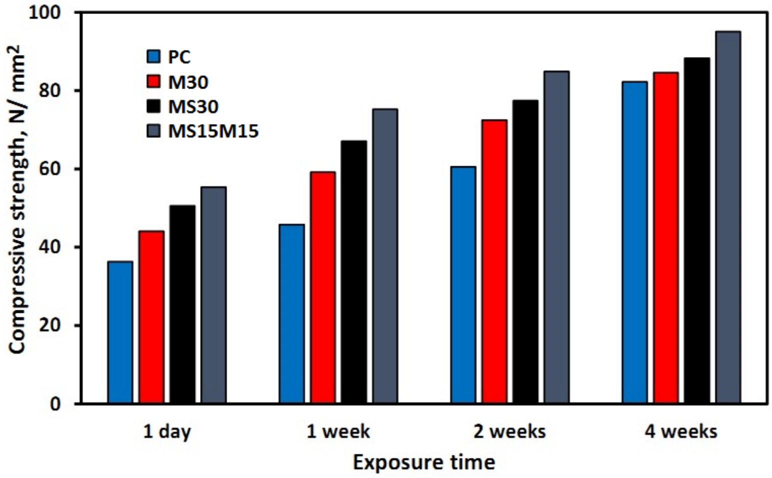


**Figure 2.** Compressive strength of the specimens with various admixtures at room temperature

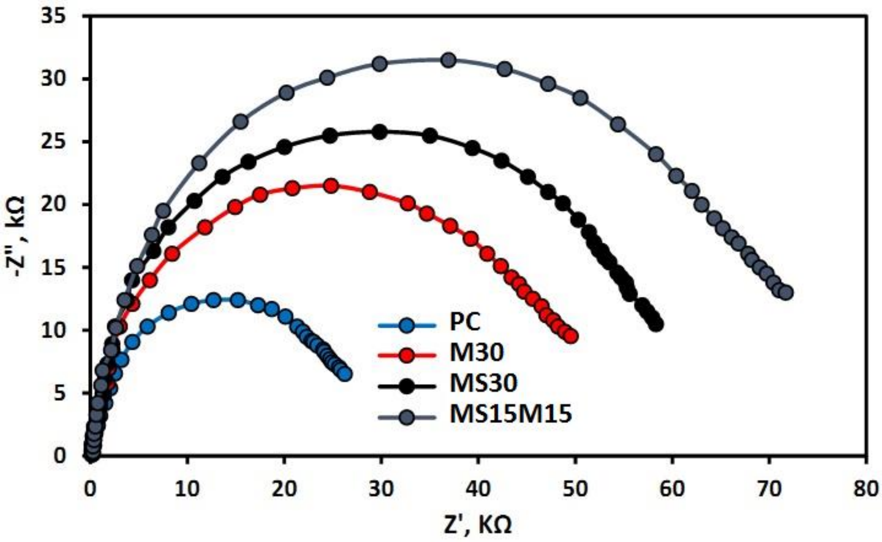


**Figure 3.** Nyquist plots achieved from carbon steel rebar in concretes with various admixtures at 3.5wt% NaCl solution in frequency range of 0.01Hz to 0.1MHzat room temperature.

EIS method has been used for studies of the corrosion behavior of carbon steel rebar embedded in concrete with various admixtures at 3.5wt% NaCl solution. The Nyquist curves achieved by EIS analysis was shown in Figure 3. An equivalent circuit model was used which is indicated in Figure 4. Where $R_s$ shows the solution resistance. $Q_c$ and $R_c$ present the capacitance and resistance of coated concrete, respectively. $R_{ct}$ and $Q_{dl}$ present the charge-transfer resistance and the double layer capacitance of carbon steel surface, respectively.

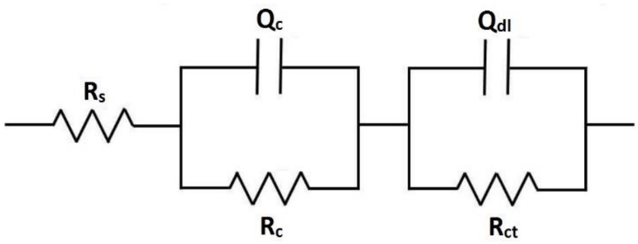


**Figure 4.** Used equivalent circuit model

The measured data are exhibited in Table 5. As indicated in table 5, with suitable replacement of MS and metakaolin in the PC cement, $R_C$ enhances and $Q_C$ reduces, which indicates an increase in the corrosion resistance, stability and thickness of the passive film on the carbon steel embedded in concrete [18]. The MS30 has a pozzolanic reaction by the $Ca(OH)_2$ crystals and create a monolithic, dense and insoluble gel of calcium hydroxide [19]. Besides, the metakaolin can form a strong adhesion to hydrated cement due to high surface area which causes a better prevention of growth of the calcium hydroxide. The mineral mixtures fill up the tiny cracks and capillary pores and then shrink the cement structures. These agents enhance the corrosion resistance of carbon steel rebars in corrosive solutions [20]. Moreover, it was observed that $Q_{dl}$ was higher than $Q_c$ in all specimens which approve formation of thin passive layer and the double layer in the interfaces has a higher capacitive behavior.

**Table 5.** The achieved data of fitting Nyquist plots for carbon steel rebar embedded in concrete with various admixtures exposed to 3.5wt% NaCl solution.

| Mixtures | $R_s$ (Ω) | $R_c$ (kΩ) | $Q_c$(μF cm$^{-2}$) | $R_{ct}$ (kΩ) | $Q_{dl}$(μF cm$^{-2}$) |
|---|---|---|---|---|---|
| PC | 23 | 16.7 | 4.8 | 28.4 | 7.2 |
| M30 | 24 | 33.1 | 3.3 | 51.3 | 5.2 |
| MS30 | 26 | 46.3 | 2.2 | 59.7 | 3.9 |
| MS15M15 | 27 | 52.2 | 1.1 | 74.6 | 2.3 |

Admixture efficiency ($\mu_e$) was measured by the following equation:

$$\mu_e\ (\%) = 100\times(R_{ct} - R_{ct}^*)/R_{ct} \qquad (1)$$

where $R_{ct}$ presents the charge-transfer resistance of the specimens with admixtures. $R_{ct}$* shows the charge-transfer resistance of the specimens without admixtures.

Table 6 shows the $\mu_e$ comparison of samples with various admixtures as stated in the literature. The findings indicated that the $\mu_e$ of carbon steel rebar embedded in concrete with both MS30 and metakaolin was comparable with other admixtures achieved from the literature.

**Table 6.** $\mu_e$ comparison of samples with various admixtures

| Admixtures | Environment | $\mu_e$ (%) | Ref. |
|---|---|---|---|
| Metakaolin | 3.5wt% NaCl | 30.3 | [21] |
| Granite waste dust | 5wt% NaCl | 69.2 | [22] |
| Bagasse Ash | 3.5wt% NaCl | 67.0 | [23] |
| Marble and granite waste dust | 3.5wt% NaCl | 37.5 | [24] |
| Metakaolin and MS | 3.5wt% NaCl | 61.9 | This work |

Since, electron conductive paths may be formed by adding metakaolin, hence the current density and the electrical resistivity of the carbon steel rebar had reduced. Moreover, the side effects of metakaolin on corrosion behavior of carbon steel rebar can be balanced by addition of MS, which has no remarkable effect on resistivity of metakaolin concrete but can reduce the porosity and water absorptive. In addition, MS30 can optimize the metakaolin dispersion in concrete structure. Thus, the addition of the metakaolin and MS simultaneously help to promote concrete structures that reduce the absorption of chloride ion and water on the carbon steel rebar. Also, it can be known as an appropriate solution to decrease negative effects for addition of any mineral additives.

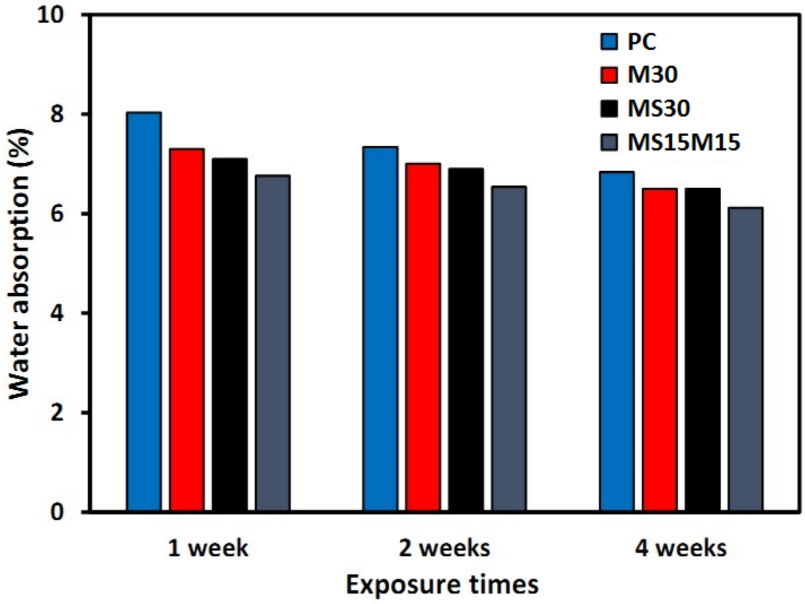


**Figure 5.** Water absorption from partial replacement of PC with MS and metakaolin after one, two and four weeks exposed to 3.5wt% NaCl solution at room temperature

The water absorption from partial replacement of PC with MS30 and metakaolin after one, two and four weeks exposed to 3.5wt% NaCl solution are shown in Fig. 5.As indicated, all the samples with metakaolin and MS exhibit a decrease in water absorption when exposure time increases. This means that metakaolin and MS additives in PC can reduce the water absorption of the samples after being exposed to a salty environment. Therefore, water absorption of the concrete samples reduces when the mixes contain both MS and metakaolin. Additionally, it can be determined that MS and metakaolin admixtures in PC had no important effect on the water absorption compared by other properties.

Although the presence of water is required in the early stages of corrosion, adsorption and of permeability the specimens do not play an important role in the corrosion resistance of carbon steel rebar which is consistent with previous studies [25]. In fact, it is important to consider the release of chloride and oxygen for anodic and cathodic reactions, respectively.

Figure 6 reveals the surface morphology of carbon steel rebars and corrosion products on steel embedded in concrete specimens with various admixtures. As presented in SEM images, the corroded areas observed on the steel rebars for MS15M15 sample were not intense and pitting corrosion on the carbon steel rebars was notably lower than the other samples. Therefore, based on these results, a suitable concrete mix in contrast to corrosion should be provided with cement replacement by MS and metakaolin.

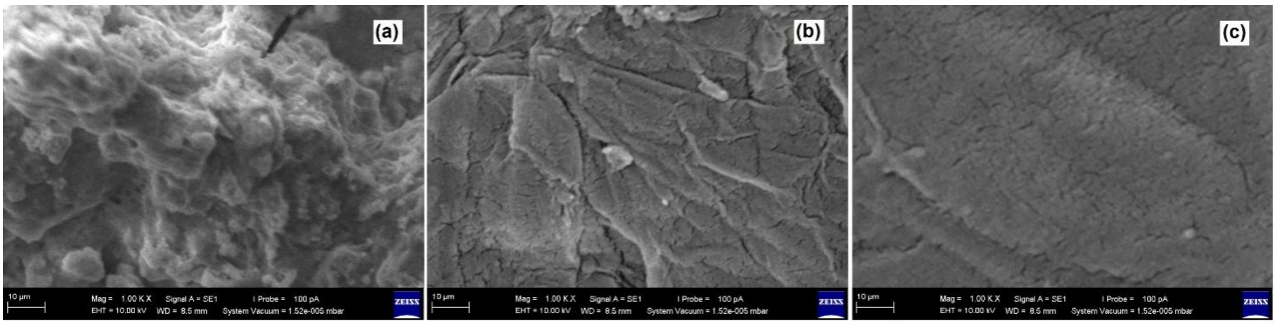


**Figure 6.** SEM images of carbon steel embedded in concrete specimen with various admixtures (a) PC (b) Metakaolin (c) MS and metakaolin

Figure 7 indicates polarization curves of carbon steel embedded in concrete (MS15M15) in electrolyte solution with and without DPLE inhibitor after immersion time of 24 h at temperature of 25°C. Figure 7 reveals that both cathodic and anodic reactions of carbon steel corrosion are inhibited in the presence of DPLE in 3.5 wt% NaCl solution and the inhibition effect enhances with increasing the DPLE concentration. Electrochemical parameters attained by Tafel extrapolation, corrosion current density ($I_{corr}$), Tafel slopes ($\beta_a$ and $\beta_c$) and corrosion potential ($E_{corr}$) are presented in Table 7. The inhibition efficiencies of DPLE in saline solution are also shown in Table 7. The inhibition efficiency (IE) is calculated as:

$$IE(\%) = 100\times(I_{corr} - I_{corr}^{*})/ I_{corr} \quad (2)$$

where $I_{corr}$ and $I_{corr}^{*}$ are the values of corrosion current density with and without DPLE inhibitor, respectively. Increasing the inhibitor concentration reduces the corrosion current density.

The presence of DPLE inhibitor results in a small shift of the $E_{corr}$ toward active directions in comparison with the result obtained without inhibitor. The reduction in both the cathodic and anodic densities in saline solution shows that the DPLE inhibitor suppresses both the cathodic and anodic reactions which can be related to the presence of DPLE on carbon steel surfaces.

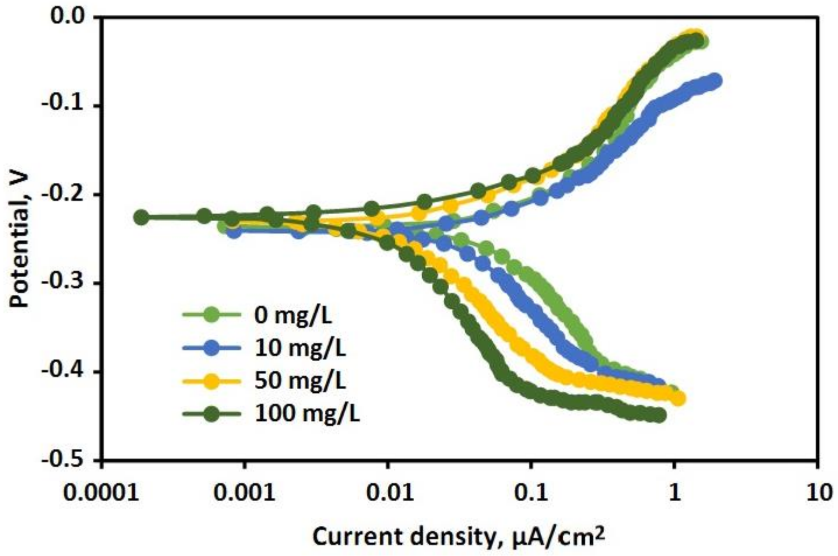


**Figure 7**. Polarization curves of carbon steel rebar embedded in concrete (MS15M15) in electrolyte solution with and without DPLE inhibitor after immersion time of 24 h at temperature of 25°

Furthermore, in the presence of DPLE inhibitor the values of $\beta_a$ were altered more than $\beta_c$ values which indicate that the anodic reactions were controlled mainly than cathodic reactions in all concentrations. Given that the changes in $\beta_a$ values are more considerable than the change in $\beta_c$ values, it is shown that the DPLE inhibition effect is anodic dominating. These results propose that the considered inhibitor acts as a mixed-type inhibitor [26]. It is found that the EI increases with increasing inhibitor concentration. Highest inhibition efficiency obtained in 3.5 wt% NaCl solution with 100 mg/L of DPLE. Moreover, these findings reveal that $I_{corr}$ reduces with increasing DPLE inhibitor concentration, and the addition of DPLE cannot vary the values of $\beta_c$ and $E_{corr}$. All the results achieved from EIS and polarization studies are in good accordance with together.

**Table 7.** Electrochemical parameters attained by Tafel extrapolation

| DPLE contents | $I_{corr}$ (µA/cm$^2$) | $E_{corr}$(V) | $\beta_a$ (mV/decade) | $\beta_c$ (mV/decade) | IE (%) |
|---|---|---|---|---|---|
| 0 mg/L | 0.089 | -0.248 | 104 | 176 | - |
| 10 mg/L | 0.052 | -0.242 | 65 | 155 | 41.5 |
| 50 mg/L | 0.038 | -0.238 | 53 | 151 | 57.3 |
| 100 mg/L | 0.011 | -0.231 | 55 | 163 | 87.6 |

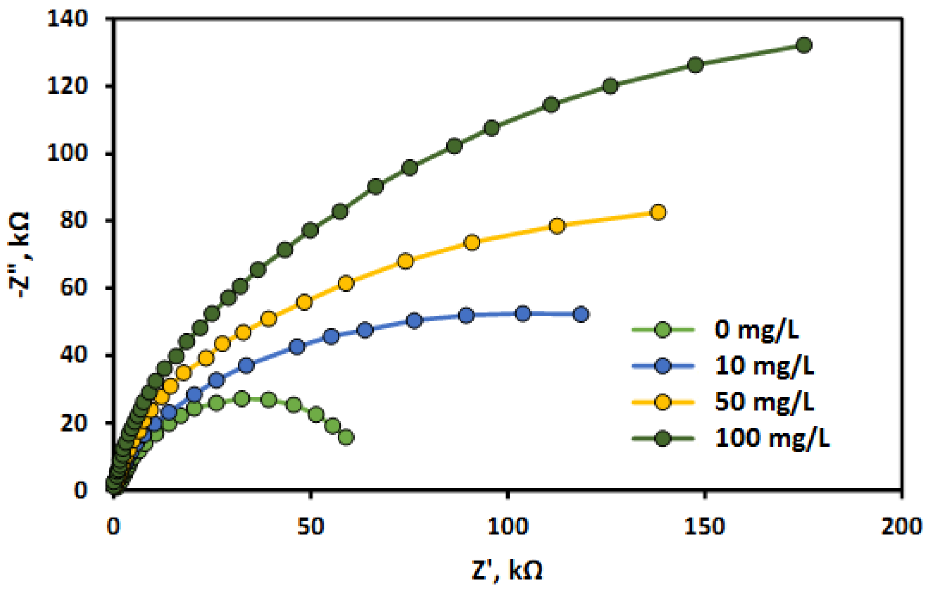


**Figure 8.** EIS diagrams of carbon steel rebar in concrete (MS15M15) in electrolyte solution with and without DPLE inhibitor after immersion time of 24 h at room temperature

The Nyquist plots of carbon steel rebar in concrete (MS15M15) after exposure time of 24 h with different concentrations of DPLE inhibitor is revealed in Figure 8.

As shown, the sample were immersed to the 3.5wt% NaCl solution without inhibitor indicated one whole half semicircle loop and the sample that was exposed to the 3.5wt% NaCl solution with DPLE revealed an incomplete semicircle loops and larger radius than the one without inhibitor, indicating a superior corrosion resistance behavior of the inhibitor. When the inhibitor concentration increased, the semi-circle loop diameter increased because of the formation of passive layer or adsorption of DPLE in anodic sites of carbon steel and therefore reduced the iron dissolution [27]. The passive film decreased the attack of chloride ions on the surface of steel rebar and efficiently controlled the pitting corrosion.

The best fitting parameters based on the equivalent circuit (Fig. 4) are summarized in Table 8. As shown, the $R_{ct}$ values were significantly increased from 74.6 kΩ to 474.2 kΩ, as concentration of DPLE inhibitor increased in the 3.5wt% NaCl solution which showed that the presence of DPLE caused the enhance the corrosion resistance of the carbon steel.

**Table 8.** EIS parameters derived from the fitting equivalent circuit for carbon steel rebar into the 3.5 wt% NaCl solution with different concentration of DPLE inhibitor after 24 h exposure time

| Inhibitor amounts | $R_s$ (Ω) | $R_c$(kΩ) | $Q_c$(μF cm$^{-2}$) | $R_{ct}$ (kΩ) | $Q_{dl}$(μF cm$^{-2}$) |
|---|---|---|---|---|---|
| 0 mg/L | 27 | 52.2 | 1.1 | 74.6 | 2.3 |
| 10 mg/L | 28 | 114.5 | 0.9 | 210.4 | 1.4 |
| 50 mg/L | 31 | 136.5 | 0.5 | 275.8 | 0.9 |
| 100 mg/L | 29 | 297.4 | 0.4 | 474.2 | 0.6 |

## 4. CONCLUSIONS

In this study, the simultaneous effect of mineral admixtures and a green inhibitor on electrochemical corrosion behavior of carbon steel embedded in concrete containing metakaolin and MS30 were investigated in 3.5wt% NaCl solution. The compressive strengths exhibited that simultaneously replacement of MS30 and metakaolin in PC improved the mechanical properties of samples. Electrochemical impedance spectroscopy technique and polarization measurement, water absorption test and compressive strength were used to assess the corrosion resistance of carbon steel embedded in concrete. The electrochemical results revealed that the MS15M15 concrete containing both MS30 and metakaolin had higher corrosion potential and resistance than the other samples. The water absorption results exhibited that the MS15M15 specimen significantly influence on mobility and penetration of water and chloride ions. The morphology characterization of the surface of carbon steel rebar showed that slight pits and low corrosion products appeared on the surface of MS15M15 sample which was according to the results achieved from electrochemical tests. The polarization results revealed that the DPLE was a mixed-type inhibitor that improved the corrosion resistant performance in carbon steel rebar considerably with formation of an organic layer to prevent both cathodic and anodic corrosion reactions. These findings exhibited that mineral admixtures and DPLE led to a decrease of hydroxide ion contents which caused an enhancement in passivation of carbon steel rebar and an enhanced corrosion resistance.

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